

=> d his ful

(FILE 'REGISTRY' ENTERED AT 13:34:18 ON 16 FEB 2006)

DEL HIS Y
ACT ROBERTCL147/A

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L1          STR
L2 (        312)SEA SSS FUL L1
L3          STR
L4          230 SEA SUB=L2 SSS FUL L3

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ACT ROBERTEX1718/A

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L5          STR
L6 (        312)SEA SSS FUL L5
L7          STR
L8 (        230)SEA SUB=L6 SSS FUL L7
L9          STR
L10         38 SEA SUB=L8 SSS FUL L9

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FILE 'CAPLUS' ENTERED AT 13:34:51 ON 16 FEB 2006

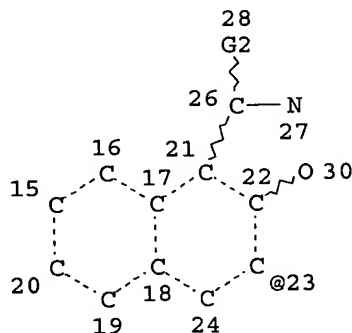
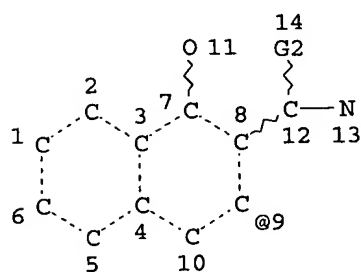
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L11         63 SEA ABB=ON PLU=ON L4
L12         16 SEA ABB=ON PLU=ON L10
           D SCAN TI

```

=> d que sta l4

L1 STR



G1 29

```

VAR G1=9/23
VAR G2=CY/AK
NODE ATTRIBUTES:
NSPEC  IS RC      AT 13
NSPEC  IS RC      AT 27
CONNECT IS E3     RC AT 13
CONNECT IS E3     RC AT 27
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

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GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS 29

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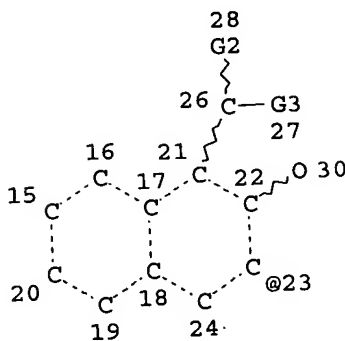
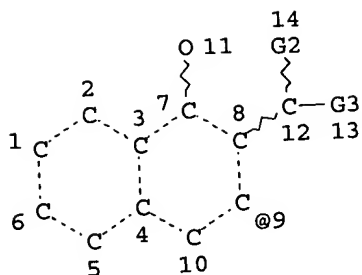
STEREO ATTRIBUTES: NONE
L2 (        312)SEA FILE=REGISTRY SSS FUL L1
L3          STR

```

Robert Shiao 10/757,581

G1 29

Hy @34



Ak~N~Ak
35 @36 37

Cb~N~Cb
38 @39 40

Cb~N~Ak
41 @42 43

VAR G1=9/23
VAR G2=CY/AK
VAR G3=34/36/39/42
NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
GGCAT IS MCY SAT AT 34
DEFAULT ECLEVEL IS LIMITED
ECOUNT IS E1 N X1 O AT 34

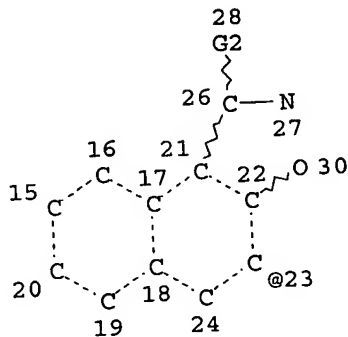
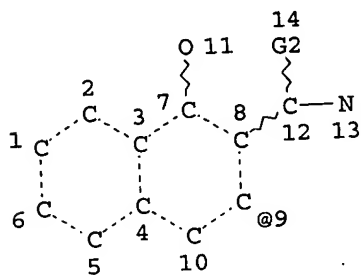
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RSPEC I
NUMBER OF NODES IS 39

STEREO ATTRIBUTES: NONE
L4 230 SEA FILE=REGISTRY SUB=L2 SSS FUL L3

100.0% PROCESSED 312 ITERATIONS
SEARCH TIME: 00.00.01

230 ANSWERS

=> d que sta l10
L5 STR



G1 29

VAR G1=9/23
VAR G2=CY/AK
NODE ATTRIBUTES:
NSPEC IS RC AT 13
NSPEC IS RC AT 27

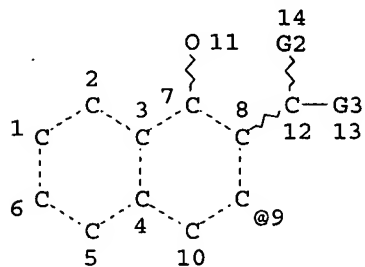
CONNECT IS E3 RC AT 13
 CONNECT IS E3 RC AT 27
 DEFAULT MLEVEL IS ATOM
 DEFAULT ECLEVEL IS LIMITED

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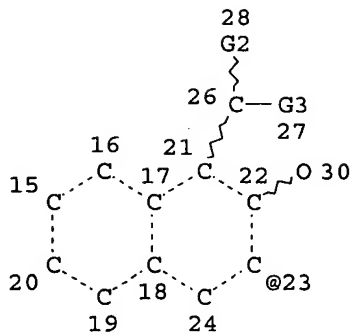
RSPEC I
 NUMBER OF NODES IS 29

STEREO ATTRIBUTES: NONE

L6 (312)SEA FILE=REGISTRY SSS FUL L5
 L7 STR



G1 29



Hy @34

Ak~N~Ak
 35 @36 37

Cb~N~Cb
 38 @39 40

Cb~N~Ak
 41 @42 43

VAR G1=9/23
 VAR G2=CY/AK
 VAR G3=34/36/39/42

NODE ATTRIBUTES:

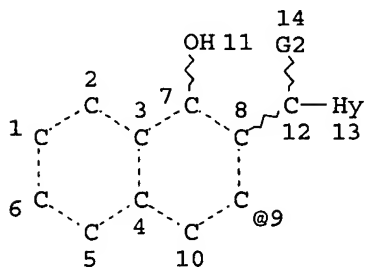
DEFAULT MLEVEL IS ATOM
 GGCAT IS MCY SAT AT 34
 DEFAULT ECLEVEL IS LIMITED
 ECOUNT IS E1 N X1 O AT 34

GRAPH ATTRIBUTES:

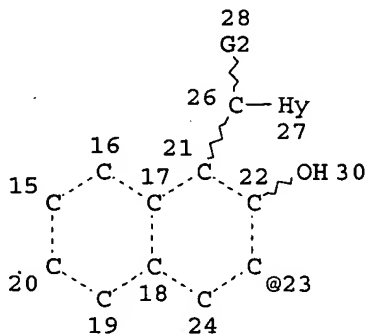
RSPEC I
 NUMBER OF NODES IS 39

STEREO ATTRIBUTES: NONE

L8 (230)SEA FILE=REGISTRY SUB=L6 SSS FUL L7
 L9 STR



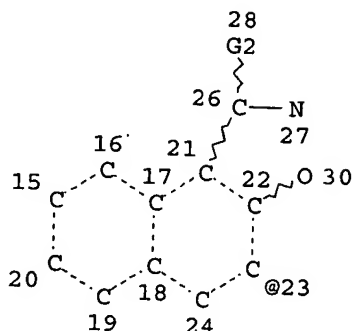
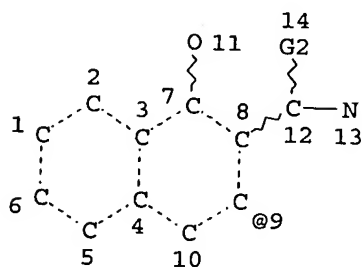
G1 29



<http://www.cas.org/ONLINE/UG/regprops.html>

=> d que stat l4

L1 STR



G1 29

VAR G1=9/23

VAR G2=CY/AK

NODE ATTRIBUTES:

NSPEC IS RC AT 13

NSPEC IS RC AT 27

CONNECT IS E3 RC AT 13

CONNECT IS E3 RC AT 27

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

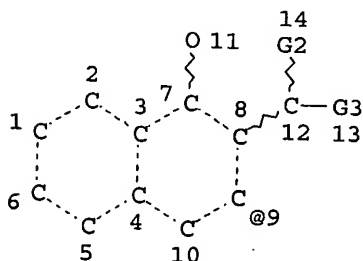
RSPEC I

NUMBER OF NODES IS 29

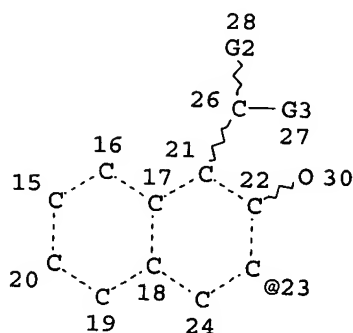
STEREO ATTRIBUTES: NONE

L2 (312)SEA FILE=REGISTRY SSS FUL L1

L3 STR



G1 29



Hy @34

Ak~N~Ak
35 @36 37

Cb~N~Cb
38 @39 40

Cb~N~Ak
41 @42 43

VAR G1=9/23

VAR G2=CY/AK

VAR G3=34/36/39/42

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

GGCAT IS MCY SAT AT 34

DEFAULT ECLEVEL IS LIMITED

ECOUNT IS E1 N X1 O AT 34

RSPEC I

NUMBER OF NODES IS 39

STEREO ATTRIBUTES: NONE

L4 230 SEA FILE=REGISTRY SUB=L2 SSS FUL L3

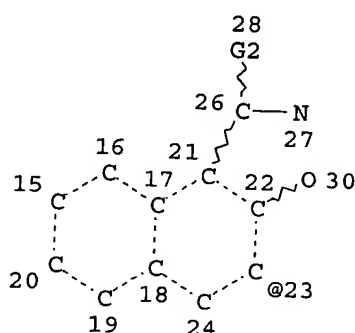
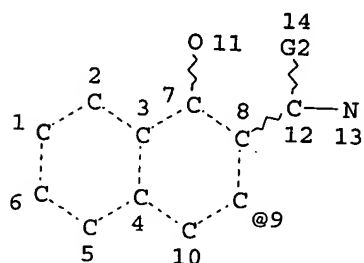
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100.0% PROCESSED      312 ITERATIONS
SEARCH TIME  00.00
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SEARCH TIME: 00.00.01

230 ANSWERS

```
=> d que stat 110
```

L5 STR



G1 29

VAR G1=9/23

```
VAR G2=CY/AK
```

NODE ATTRIBUTES:

NSPEC IS RC AT 13

NSPEC	IS	RC	AT	27
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CONNECT IS E3 RC AT 13

CONNECT IS E3 RC AT 27

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RSPEC I

NUMBER OF NODES IS 29

STEREO ATTRIBUTES: NONE

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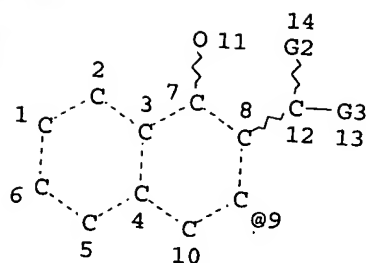
L6 ( 312)SEA FILE=REGISTRY SSS FUL L5
L7 STR

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L7 STR

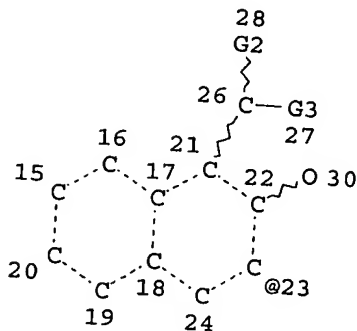
↳ claim 147

Robert Shiao 10/757,581



G1 29

Hy @34



Ak~N~Ak
35 @36 37

Cb~N~Cb
38 @39 40

Cb~N~Ak
41 @42 43

VAR G1=9/23

VAR G2=CY/AK

VAR G3=34/36/39/42

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

GGCAT IS MCY SAT AT 34

DEFAULT ECLEVEL IS LIMITED

ECOUNT IS E1 N X1 O AT 34

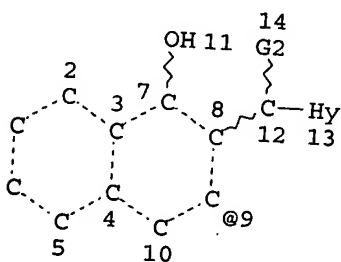
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RSPEC I

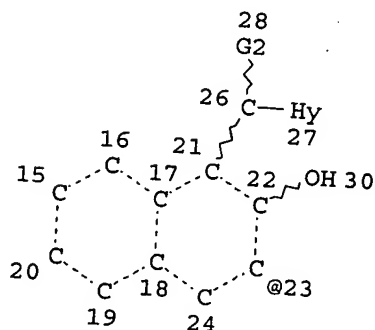
NUMBER OF NODES IS 39

STEREO ATTRIBUTES: NONE

L8 (230)SEA FILE=REGISTRY SUB=L6 SSS FUL L7
L9 STR



G1 29



R G1=9/23

R G2=CY/AK

DE ATTRIBUTES:

FAULT MLEVEL IS ATOM

CAT IS MCY SAT AT 13

CAT IS MCY SAT AT 27

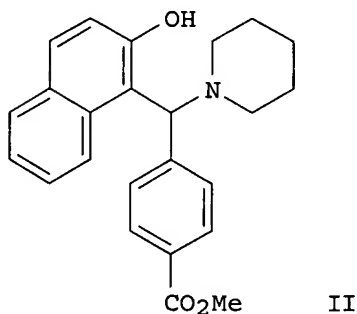
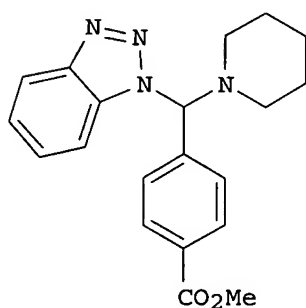
FAULT ECLEVEL IS LIMITED

UNT IS E1 N E1 O AT 13

UNT IS E1 N E1 O AT 27

PH ATTRIBUTES:

L3 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1999:436700 CAPLUS
 DOCUMENT NUMBER: 131:243022
 TITLE: Amino(hetero)arylmethylation of phenols with
 N-[α -amino(hetero)arylmethyl]benzotriazoles
 AUTHOR(S): Katritzky, Alan R.; Abdel-Fattah, Ashraf A. A.;
 Tymoshenko, Dmytro O.; Belyakov, Sergei A.; Ghiviriga,
 Ion; Steel, Peter J.
 CORPORATE SOURCE: Center for Heterocyclic Compounds Department of
 Chemistry, University of Florida, Gainesville, FL,
 32611-7200, USA
 SOURCE: Journal of Organic Chemistry (1999), 64(16), 6071-6075
 CODEN: JOCEAH; ISSN: 0022-3263
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 131:243022
 GI



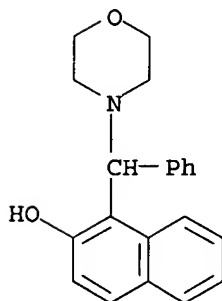
AB N-[α -Amino(hetero)arylmethyl]benzotriazoles derived from a variety
 of (hetero)aromatic aldehydes were reacted with sodium phenolates to afford
 amino(hetero)arylmethylated phenols in high yields. Thus, the
 benzotriazolylbenzoate I was prepared by condensation of benzotriazole,
 4-(HCO)C₆H₄CO₂Me, and piperidine; reaction of I with β -naphthol
 sodium salt in refluxing toluene containing a phase-transfer catalyst,
 dibenzo-18-crown-6, gave 82% [(methoxycarbonyl)benzyl]naphthol II.

IT 24685-08-3P 244184-48-3P

RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of (aminobenzyl)phenols and (aminoheterarylmethyl)phenols by
 condensation of phenols with (aminobenzyl)benzotriazoles and
 (aminoheterarylmethyl)benzotriazoles)

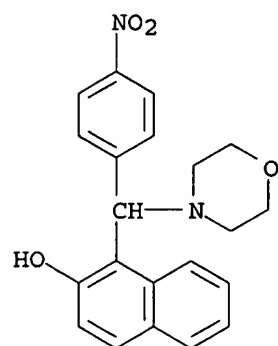
RN 24685-08-3 CAPLUS

CN 2-Naphthalenol, 1-(4-morpholinylphenylmethyl)- (9CI) (CA INDEX NAME)



RN 244184-48-3 CAPLUS

CN 2-Naphthalenol, 1-[4-morpholinyl(4-nitrophenyl)methyl]- (9CI) (CA INDEX NAME)



L6 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:698228 CAPLUS

DOCUMENT NUMBER: 128:13099

TITLE: A tandem aminoalkylation of aldehydes; application to the synthesis of substituted phenols and naphthols

AUTHOR(S): Saidi, Mohammad; Khalaji, Hamid R.

CORPORATE SOURCE: Dep. Chem., Sharif Univ. Technol., Tehran, 11365-9516, Iran

SOURCE: Journal of Chemical Research, Synopses (1997), (9), 340-341

CODEN: JRPSDC; ISSN: 0308-2342

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 128:13099

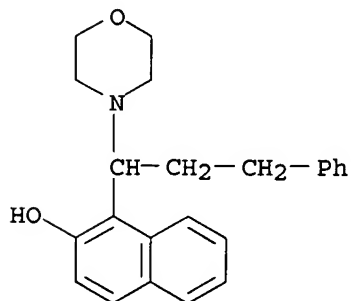
AB Treatment of a protected salicylaldehyde and 2-hydroxy-1-naphthaldehyde with (trimethylsilyl)dialkylamines and various nucleophiles in a 5 M di-Et ether solution of lithium perchlorate gives a variety of N,N-dialkylaminophenols and 1-(N,N-dialkylamino)-2-naphthols in short reaction times and in good yields.

IT 198987-08-5P 198987-10-9P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

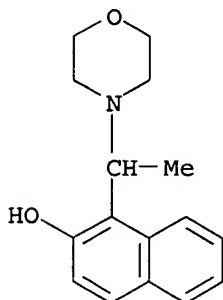
RN 198987-08-5 CAPLUS

CN 2-Naphthalenol, 1-[1-(4-morpholinyl)-3-phenylpropyl]- (9CI) (CA INDEX NAME)



RN 198987-10-9 CAPLUS

CN 2-Naphthalenol, 1-[1-(4-morpholinyl)ethyl]- (9CI) (CA INDEX NAME)



L3 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:698228 CAPLUS

DOCUMENT NUMBER: 128:13099

TITLE: A tandem aminoalkylation of aldehydes; application to the synthesis of substituted phenols and naphthols

AUTHOR(S): Saidi, Mohammad; Khalaji, Hamid R.

CORPORATE SOURCE: Dep. Chem., Sharif Univ. Technol., Tehran, 11365-9516, Iran

SOURCE: Journal of Chemical Research, Synopses (1997), (9), 340-341

CODEN: JRPSDC; ISSN: 0308-2342

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 128:13099

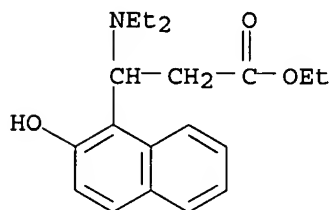
AB Treatment of a protected salicylaldehyde and 2-hydroxy-1-naphthaldehyde with (trimethylsilyl)dialkylamines and various nucleophiles in a 5 M di-Et ether solution of lithium perchlorate gives a variety of N,N-dialkylaminophenols and 1-(N,N-dialkylamino)-2-naphthols in short reaction times and in good yields.

IT 198986-99-1P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 198986-99-1 CAPLUS

CN 1-Naphthalenepropanoic acid, β -(diethylamino)-2-hydroxy-, ethyl ester
(9CI) (CA INDEX NAME)



REFERENCE COUNT:

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1993:254274 CAPLUS

DOCUMENT NUMBER: 118:254274

TITLE: Photo- and thermochromic Mannich bases. 1. Mannich bases from 3,5-di-tert-butyl-4-hydroxybenzaldehyde and 2-naphthols

AUTHOR(S): Komissarov, V. N.; Ukhin, L. Yu.; Kharlanov, V. A.; Lokshin, V. A.; Bulgarevich, E. Yu.; Minkin, V. I.; Filipenko, O. S.; Novozhilova, M. A.; Aldoshin, S. M.; Atovmyan, L. O.

CORPORATE SOURCE: Inst. Chem. Phys., Chernogolovka, 142432, Russia
SOURCE: Izvestiya Akademii Nauk, Seriya Khimicheskaya (1992), (10), 2389-99

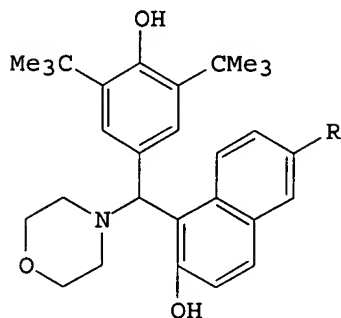
CODEN: IASKEA; ISSN: 1026-3500

DOCUMENT TYPE: Journal

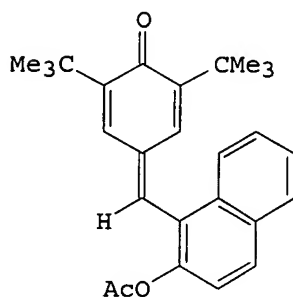
LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 118:254274

GI



I



II

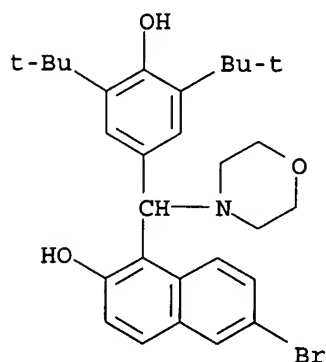
AB Mannich bases I (R = H, Br) synthesized from 3,5-di-tert-butyl-4-hydroxybenzaldehyde and 2-naphthols are photo- and thermochromic in solns. The study of acetyl derivative (II) of methylenequinone modeling the product of photo- and thermochromic conversions proved that the color changes of solns. of Mannich bases is due to the reversible dissociation into colored methylenequinones and morpholine. Mol. structure and dissociation mechanism are discussed in terms of x-ray crystallog. anal. of I (R = H).

IT 147711-20-4P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and photo- and thermochromism of)

RN 147711-20-4 CAPLUS

CN 2-Naphthalenol, 1-[[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-4-morpholinylmethyl]-6-bromo- (9CI) (CA INDEX NAME)



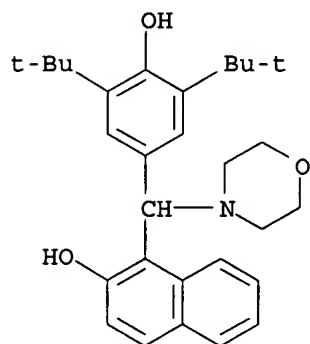
IT 147711-19-1P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation, photo- and thermochromism, and crystal and mol. structure of)

RN 147711-19-1 CAPLUS

CN 2-Naphthalenol, 1-[[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-4-morpholinylmethyl]- (9CI) (CA INDEX NAME)



L13 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1974:108438 CAPLUS

DOCUMENT NUMBER: 80:108438

TITLE: Synthesis of Δ^2 -pyrazolines and 1-naphthols

AUTHOR(S): Fateen, Abdel K.; Ali, Morsy M.

CORPORATE SOURCE: Fac. Sci., Ain Shams Univ., Cairo, Egypt

SOURCE: Egyptian Journal of Chemistry (1972), 15(4), 329-36

CODEN: EGJCA3; ISSN: 0449-2285

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

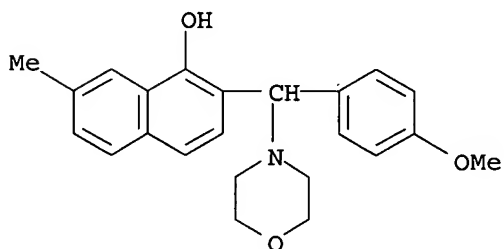
AB The naphthopyrazolines I (R = Ph, p-MeOC₆H₄; R₁ = H, Me; R₂ = Me, Ph; R₃ = Ac, Ph) were prepared by reaction of the tetralones II with PhNHNH₂ or H₂NNH₂ in HOAc. The naphthols III (R = Ph, p-MeOC₆H₄; R₁ = H, Me; R₂ = morpholino, piperidino) were prepared by bromination of II followed by reaction with amines.

IT 52159-83-8P 52159-86-1P 52159-87-2P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

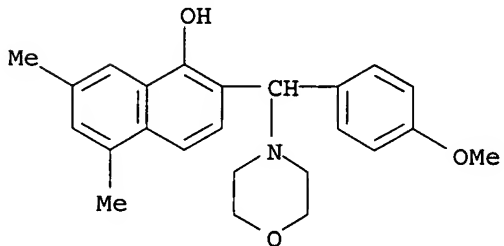
RN 52159-83-8 CAPLUS

CN 1-Naphthalenol, 2-[(4-methoxyphenyl)-4-morpholinylmethyl]-7-methyl- (9CI)
(CA INDEX NAME)



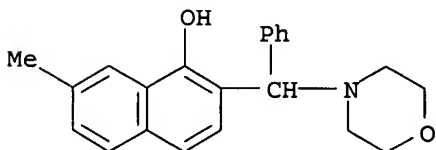
RN 52159-86-1 CAPLUS

CN 1-Naphthalenol, 2-[(4-methoxyphenyl)-4-morpholinylmethyl]-5,7-dimethyl- (9CI) (CA INDEX NAME)



RN 52159-87-2 CAPLUS

CN 1-Naphthalenol, 7-methyl-2-(4-morpholinylphenylmethyl)- (9CI) (CA INDEX NAME)



L17 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1970:31500 CAPLUS

DOCUMENT NUMBER: 72:31500

TITLE: Synthesis of 2,3-arenophenalenium cations. A new synthetic use for Mannich bases

AUTHOR(S): Seshadri, S.; Cherian, A. L.; Pandit, P. Y.

CORPORATE SOURCE: Dep. Chem. Technol., Univ. Bombay, Bombay, India

SOURCE: Indian Journal of Chemistry (1969), 7(11), 1080-3

CODEN: IJOCAP; ISSN: 0019-5103

DOCUMENT TYPE: Journal

LANGUAGE: English

GI For diagram(s), see printed CA Issue.

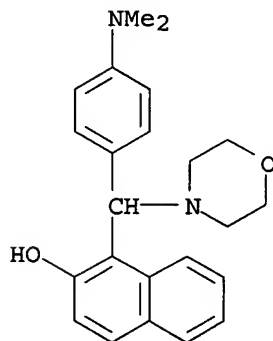
AB A synthesis of 2,3-arenophenalenium perchlorates (e.g. I), based on a novel reaction of Mannich bases derived from β -naphthol by condensation with an aromatic aldehyde and morpholine, is given. The O-Me derivs. of the Mannich bases are subjected to a cyclo-dehydrogenation reaction to give the desired phenalenium derivs. in good yields. The absorption spectra of the new compds. are discussed.

IT 24685-05-0P 24685-06-1P 24685-07-2P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

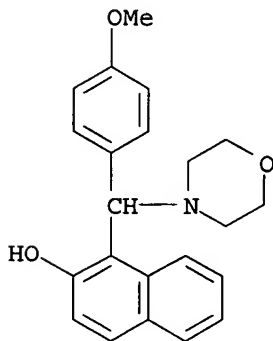
RN 24685-05-0 CAPLUS

CN 2-Naphthol, 1-[p-(dimethylamino)- α -morpholinobenzyl]- (8CI) (CA INDEX NAME)



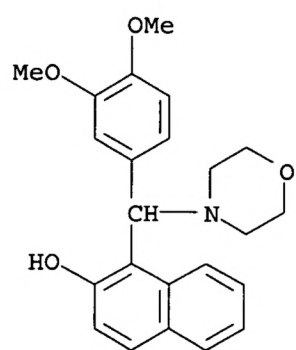
RN 24685-06-1 CAPLUS

CN 2-Naphthol, 1-(p-methoxy- α -morpholinobenzyl)- (8CI) (CA INDEX NAME)



RN 24685-07-2 CAPLUS

CN 2-Naphthol, 1-(α -morpholinoveratryl)- (8CI) (CA INDEX NAME)



=>

ACCESSION NUMBER: 1959:7062 CAPLUS
DOCUMENT NUMBER: 53:7062
ORIGINAL REFERENCE NO.: 53:1346c-i,1347a-f
TITLE: Benzacridines. I. Syntheses and reactions of
5,6-dihydrobenz[c]acridines
AUTHOR(S): Bell, Vernon L.; Cromwell, Norman H.
CORPORATE SOURCE: Univ. of Nebraska, Lincoln
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AB A convenient synthesis was developed to prepare a number of substituted dihydrobenz[c]acridines and benz[c]acridines. 5,6-Dimethylbenz[c]acridine (I) was obtained through a Wagner-Meerwein rearrangement brought about by an α -elimination of HBr from 6-bromo-5,5-dimethyl-5,6-dihydrobenz[c]acridine (II). The ultraviolet absorption spectra of these new compds. are reported and compared. 4 - Methyl - 4 - phenylpentanethiomorpholide (246 g.) refluxed 48 hrs. with 1.51. concentrated HCl, cooled, extracted with 1 l. C₆H₆, the organic layer extracted with 25% NaOH, the

alkaline solution acidified, and the aqueous mixture extracted with C₆H₆ and concentrated gave

163.7 g. 4-methyl-4-phenylpentanoic acid (III), b_{0.8} 140-1°. III (172 g.) heated to 65°, added in one lot to 500 g. polyphosphoric acid at 90°, the mixture stirred 3 min., placed on the steam bath, and 300 g. more polyphosphoric acid added, the mixture stirred 25 min. at 90°, cooled, poured into ice H₂O, extracted with Et₂O, washed with H₂O, 5% NaOH, H₂O, 3% AcOH, and H₂O, and distilled gave 136.9 g. 4,4-dimethyl derivative of 1-tetralone (IV), b₂ 125-31°. IV (29 g.) in 170 ml. AcOH and 25 g. o-O₂NC₆H₄CHO left 3 days at room temperature with 25 ml. 95% H₂SO₄ gave 48 g. 4,4-dimethyl-2-(o-nitrobenzal) derivative of 1-tetralone (V), m. 188-9° (AcOH). V (48 g.) dissolved while heating in 800 ml. AcOH and 80 ml. H₂O and treated hot with 20 g. Fe powder portionwise, the solution heated 15 min. after the evolution of H ceased, poured into ice H₂O, treated with 1.8 l. 33% KOH, and left overnight gave 36 g. 4,4-dimethyl-2-(o-aminobenzal) derivative of 1-tetralone (VI), m. 135-7° (alc.). Treatment of VI with picric acid gave 5,5-dimethyl-5,6-dihydrobenz[c]acridine (VII) picrate, m. 202-3°. VI in 95% alc. and 50 ml. concentrated HCl evaporated to dryness, the residue redissolved in 95% alc., treated with C, and filtered, the solution neutralized with 5% NaHCO₃ and cooled, and the product collected gave 33.6 g. free VII, m. 112-13° (aqueous Me₂CO); picrate, identical with the above prepared specimen. VII was also obtained by ring closure of VI by irradiation with ultraviolet light for 30 hrs. in 95% alc. IV (6 g.), 5.05 g. isatin, 6.5 g. KOH, 10 ml. MeOH, and 6.5 ml. H₂O refluxed 8 hrs., the basic solution diluted with H₂O, extracted with Et₂O, and the

alkaline solution

acidified gave 6.5 g. 7-carboxy-5,5-dimethyl-5,6-dihydrobenz[c]acridine (VIII), m. 256.5-7.0° (dioxane.). VIII (7.6 g.) melted in a small flask at 260° for 2 hrs. until evolution of CO₂ ceased, cooled, triturated with 10% KOH, extracted with Et₂O, and evaporated gave 6.1 g. VII.

VII

(19.7 g.) in 250 ml. CCl₄ refluxed 3 hrs. with 13.5 g. N-bromosuccinimide and 0.25 g. Bz₂O₂, cooled, filtered, and extracted with 200 ml. 5% NaHCO₃, then with 300 ml. H₂O, and the CCl₄ layer evaporated gave 21 g. 6-bromo-5,5-dimethyl-5,6-dihydrobenz[c]acridine (IX), m. 145-7°. IX (0.85 g.) in 20 ml. alc. heated 1 hr. gave 0.70 g. 5,5-dimethyl-6-ethoxy-5,6-dihydrobenz[c]acridine (X), m. 96-7° (alc.); picrate, m. 197-8° (decomposition). IX (2 g.) in 25 ml. dioxane heated 1 hr. on the steam bath with 10 ml. 10% NaOH, gave 1.25 g. 5,5-dimethyl-6-hydroxy-5,6-dihydrobenz[c]acridine (XI), m. 159-60°; picrate, softened at

215° but did not melt up to 250°. IX (1 g.) in 20 ml. MeOH refluxed 3 hrs. gave 0.8 g. 5,5-dimethyl-6-methoxy-5,6-dihydrobenz[c]acridine (XII), m. 152.5-4.0° (aqueous MeOH). IX (3 g.) and 15 ml. morpholine refluxed 24 hrs., solution cooled, poured into H₂O, the solid collected, and recrystd. gave 2.6 g. 5,5-dimethyl-6-morpholino-5,6-dihydrobenz[c]acridine (XIII), m. 159-61° (alc.). IX (5 g.) and 15 ml. anhydrous Me₂NH heated 8 hrs. at 100° in a sealed tube gave 3 g. 5,5-dimethyl-6-dimethylamino-5,6-dihydrobenz[c]acridine (XIV), m. 93-5° (alc.). IX (2 g.) on heating melted to a light yellow liquid which turned to a bright red solid at 160°. Heating was continued 10 min. at 170°. The residue dissolved in warm, aqueous dioxane, neutralized, and cooled gave 1.1 g. I, m. 162-3° (Me₂CO); picrate, m. 253-4° (decomposition). o-O₂NC₆H₄CHO (15 g.) dissolved in 150 ml. AcOH and 30 g. 95% H₂SO₄ added with cooling, the mixture left 72 hrs. at room temperature with 14.6 g. α-tetralone, and the crude product collected and crystallized gave 20.9 g. 2-(o-nitrobenzal) derivative of 1-tetralone (XV), m. 121-2° (AcOH). XV (5.8 g.) in 40 ml. AcOH and 20 ml. H₂O heated to 70° and treated portion-wise with 2.5 g. Fe powder, heating continued 45 min., the solution poured on ice and H₂O, 150 ml. 33% KOH added, the mixture left overnight, the solid material collected, extracted with 200 ml. absolute alc., and H₂O added gave 3.3 g. 2-(o-amino-benzal) derivative of 1-tetralone (XVI), m. 123-4°. XVI treated with picric acid gave the picrate of 5,6-dihydrobenz[c]acridine (XVII), m. 206°. Ring closure of XVI was effected by evaporating a HCl-containing 95% alc. solution of 3.3 g. of XVI to dryness. The HCl salt dissolved in aqueous alc., treated with C, and neutralized with 5% NaHCO₃, and H₂O added gave 2.6 g. free XVII, m. 65°. The over-all yield of XVII from XV was 54%. XVII was also prepared in 88% yield using the method of Braun and Wolff (C.A. 17, 1227). The crude product m. 59-60°; treatment of an alc. solution of the HCl salt with C and neutralization with Na₂CO₃ gave pure XVII. 7-Carboxy-5,6-dihydrobenz[c]acridine (XVIII), prepared in 83% yield by the Pfitzinger-Borsche reaction, m. 250°. o-O₂NC₆H₄CHO (20 g.), 19.3 g. α-naphthol, and 12.7 g. morpholine in 17 ml. alc. left 24 hrs. under N and cooled gave 25 g. 2-(α-morpholino-o-nitrobenzyl)-1-naphthol (XIX), m. 128-9.5° (alc.). XVII was dehydrogenated to 60% benz[c]acridine (XX), m. 107-8°; picrate, m. 249°. XIX (5.5 g.) treated at 70° in 35 ml. AcOH and 15 ml. H₂O with 1.9 g. reduced Fe powder, the mixture heated an addnl. 0.5 hr. on the steam bath, poured into 200 g. ice and H₂O, treated with 100 ml. 33% KOH, and left overnight, the solid precipitate collected and extracted with

200 ml.

hot alc., the alc. solution evaporated to dryness, the residue extracted with

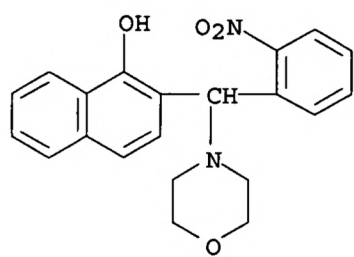
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HCl, and the acidic solution treated with C, neutralized, and recrystd. gave 10% XX, which with its picrate was identical with the above prepared specimen. The ultraviolet spectra were given for I, VI, VII, VIII, IX, X, XI, XII, XIII, XIV, XV, XVI, XVII, XVIII, and XIX. The spectra of the unsubstituted and substituted dihydrobenzacridines are quite identical, differing only slightly in both wave length and ε. The notable exception was IX, which has only a single high intensity absorption band at 220-330 mμ range and a different longer wave length fine structure than that found for other dihydrobenzacridines.

IT 6628-55-3, 1-Naphthol, 2-(α-morpholino-o-nitrobenzyl)-
(preparation of)

RN 6628-55-3 CAPLUS

CN 1-Naphthalenol, 2-[4-morpholinyl(2-nitrophenyl)methyl]- (9CI) (CA INDEX
NAME)



ACCESSION NUMBER: 1957:34766 CAPLUS

DOCUMENT NUMBER: 51:34766

ORIGINAL REFERENCE NO.: 51:6575d-i,6576a-d

TITLE: The chemistry of derivatives of 2-benzaltetralone. I.
A novel rearrangement leading to 2-substituted-1-naphthols

AUTHOR(S): Hassner, Alfred; Cromwell, Norman H.; Davis, Stanley J.

CORPORATE SOURCE: Univ. of Nebraska, Lincoln

SOURCE: Journal of the American Chemical Society (1957), 79, 230-4

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AB 1-Tetralone treated with BzH and 4% alc. KOH yielded 88% 2-benzaltetralone (I), m. 107°; the Ph and C:O groups are in trans position. I treated with alkali and 30% H2O2 in MeOH yielded 93% I oxide, m. 77-7.5° (from MeOH). I (1 g.) in 20 g. 28% HBr in glacial AcOH and 8 cc. Ac2O kept 17 hrs. at room temperature gave 85% recovered I. I (1 g.) in 26 g. glacial AcOH, 16 cc. Ac2O, and 1.7 g. H2SO4 kept 17 hrs. at room temperature gave also 85% recovered I. I (1 g.) in 15 cc. Ac2O containing 12

drops

concentrated H2SO4 kept 6 days at room temperature gave 65% recovered I and a small

amount of unidentified, red, gummy material. I (1 g.) in 12 cc. 10% NaOMe in MeOH refluxed 5 hrs. gave 85% recovered I. I heated 10-45 min. (or kept 1 day) with morpholine or piperidine gave 80-90% recovered I. Br (4.2 cc.) in 10 cc. CCl4 added with stirring to 18.4 g. I in 150 cc. CCl4 during 1 hr. gave 77% 2-bromo-2-(α -bromobenzyl)-1-tetralone (II), m. 153-4° (decomposition) (from ligroine, b. 90-100°); the residue from the filtrate gave in 1 case a small amount of solid, m. 109-9.5°, which converted to II on standing, in another case a different form of II, m. 118-19°. II (2 g.) and 10 equivs. of the appropriate liquid amine kept 8-24 hrs. under N at room temperature, the mixture

diluted with iso-Pr2O and filtered, the filtrate washed with dilute HCl and H2O and evaporated, and the liquid residue poured into cold H2O gave the corresponding 2-[α -(N-substituted-amino)benzyl]-1-naphthol (III), which was recrystd. from EtOH or ligroine (b. 60-70°) (m.p., % yield, and reaction time given): N-cyclohexyl derivative (IV), 98-100° (from ligroine, b. 60-80°), 80 (it gave tars when heated in EtOH in air), 8 hrs.; N-Me derivative (V) of IV, 140-1° (from ligroine), 91, 6 days; 2-(α -piperidinobenzyl)-1-naphthol (VI), 108-9°, 100, 8 hrs.; 2-(α -morpholinobenzyl)-1-naphthol (VII), 139° (from EtOH and ligroine), 100, 20 hrs. (HCl salt, m. 158-9°). II (1 g.) kept 1 hr. at 25° with a 10-fold excess of piperidine and filtered gave 100% piperidine-HBr; cyclohexylamine gave similarly 95% amine salt, while N-methylcyclohexylamine gave only 10% salt. II and PhNH2 heated 20 min. to 50° and then allowed to stand 2.5 days gave only 4% PhNH2.HBr. 1-C10H7OH (0.02 mole) in 2/3 its weight of absolute EtOH treated

with

1 equivalent each of BzH and the appropriate amine and the mixture kept 1-4 days

in the dark under N and seeded gave the corresponding III (m.p., % yield, and reaction time given): IV, 99-100°, 38, 1 day; V, 139.5-41°, 36, 6 hrs.; VI, 109-10°, 50, 4 days; VII, 101-3°, 50, 15 min. (at 100° and then allowed to stand 2 hrs.); VII, 131-3°, 75, 20 hrs. (at room temperature and 1.5 days in the cold); 2-[α -(N-phenylamino)benzyl]-1-naphthol, 138.5-9.5° (from EtOH or C6H6-ligroine), 85, 1 day. II (2 g.) and 7 g. 1-methylmorpholine allowed to stand 2 days gave 70% recovered II; a similar run in hot C6H6 gave 30-50% of the amine salt, but no definite

product. II refluxed with collidine gave only tars. II allowed to stand with Et₃N was recovered unchanged. II treated 3.5 days with 4-picoline at room temperature gave 15% I and largely low-melting polymeric material. II refluxed 6 hrs. with 1 equivalent KOAc in EtOH gave 67% unchanged II. II (1 g.) refluxed 4 hrs. in C₆H₆ or Me₂CO with 3 equivs. cyclohexylamine gave 30-50% crude I. I (4 g.) in 150 cc. absolute EtOH hydrogenated 20 min. at 55° and 44 lb. pressure over 0.25 g. PtO₂ gave 3.6 g.

2-benzyl-1-tetralone (VIII), colorless solid, m. 53-4° (from petr. ether). Br (4.2 g.) in 5 cc. CHCl₃ added slowly to 6.2 g. VIII in 20 cc. CHCl₃ (initially at room temperature, then with cooling) and the solution

evaporated

after 3 hrs. yielded 92% 2-Br derivative (IX) of VIII, white solid, m. 82-2.5°. IX (1 g.) allowed to stand 10 min. with 3 equivs.

morpholine, heated 2 min. on the steam bath, diluted with iso-Pr₂O, and filtered yielded 96% amine-HBr; the filtrate yielded 60% 2-PhCH₂C₁₀H₆OH

(X), m. 73-4° (from ligroine). 1-C₁₀H₇OH in PhMe treated with Na and PhCH₂Cl gave X, white crystals, m. 74-4.5° (from ligroine); it produced gradually a red coloration with the formation of a golden glimmering precipitate when treated with aqueous FeCl₃ in EtOH. A similar run

with

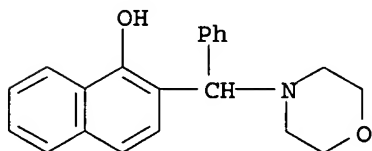
N-methylmorpholine gave only 10% HBr salt; 80% X was recovered unchanged.

IT 102474-88-4, 1-Naphthol, 2- α -morpholinobenzyl-, hydrochloride 102474-89-5, 1-Naphthol, 2- α -morpholinobenzyl-

(preparation of)

RN 102474-88-4 CAPLUS

CN 1-Naphthol, 2- α -morpholinobenzyl-, hydrochloride (6CI) (CA INDEX NAME)



● HCl

RN 102474-89-5 CAPLUS

CN 1-Naphthol, 2- α -morpholinobenzyl- (6CI) (CA INDEX NAME)

